

# Etch performance of Ar/N<sub>2</sub>/F<sub>2</sub> for CVD/ALD chamber clean

## EXECUTIVE OVERVIEW

A new F<sub>2</sub> gas mixture was evaluated as a substitute for conventional cleaning gases such as, NF<sub>3</sub>, C<sub>2</sub>F<sub>6</sub>, and CF<sub>4</sub>, in a CVD chamber. The new mixture was compatible with the equipment used and improvement was seen in the etch rate as well as a reduction in the amount of gas needed to complete the clean.

F<sub>2</sub> gas mixtures offer ideal properties as chamber cleaning gas: low dissociation energy and high reactivity, which leads to superior efficiency and ease of abatement. In this work, a new F<sub>2</sub> gas mixture with a combination ratio of 10% Ar, 20% F<sub>2</sub> and 70% N<sub>2</sub> was used to obtain a maximum of 20% fluorine in inert gases. This mixture has been evaluated as a candidate to replace conventional cleaning gases such as NF<sub>3</sub>, C<sub>2</sub>F<sub>6</sub>, and CF<sub>4</sub>. The tested Ar/N<sub>2</sub>/F<sub>2</sub> mixture showed improvements in both parameters, cleaning up to 27% faster, and requiring 96% less gas than when using NF<sub>3</sub>. The etch-rate performance of the Ar/N<sub>2</sub>/F<sub>2</sub> gas mixture was combined with etch nonuniformity values of ±3% (1σ) on SiO<sub>2</sub>, and ±8% (1σ) on Si<sub>3</sub>N<sub>4</sub>, respectively. Additionally, amorphous silicon (a-Si) was etched completely and uniformly. The particle performance data showed an average of 14 particle adders (0.25μm), indicating that no significant particle contamination was induced by the process and that Ar/N<sub>2</sub>/F<sub>2</sub> can be used as a highly clean and efficient etching gas.

## Cleaning strategies for deposition chambers

CVD and ALD depositions usually cover the surface of the substrate and the chamber walls. Regular residual removal is necessary to obtain stable and repeatable deposition results with uniform surfaces at acceptable particle levels [1]. Chamber cleaning gases such as, CF<sub>4</sub> and C<sub>2</sub>F<sub>6</sub>, have been used for quite some time [2-3]. However, the strict requirements to sustain stable processes have led to more frequent chamber cleans. NF<sub>3</sub> has emerged as the main gas for cleaning 300mm tools equipped with remote plasma source (RPS) systems; it shows significantly shorter clean times compared to C<sub>x</sub>F<sub>y</sub>-based cleans [5,6]. While NF<sub>3</sub> meets the above mentioned requirements and is considered to be easily integrated into the fab, the gas is relatively expensive.

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The Ar/N<sub>2</sub>/F<sub>2</sub> used in this studied was delivered from conventional stainless steel cylinder gas bottles. Mixtures of 20% F<sub>2</sub> are easily available for thermal cleaning processes [7]. Our approach differs from on-site fluorine generation that utilizes hydrogen fluoride (HF) as feed material [8-10]. Since the deposition, etching, and selective removal of silicon-based dielectric materials [11] are very important process applications, we have used SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub> layers on Si for the tests.

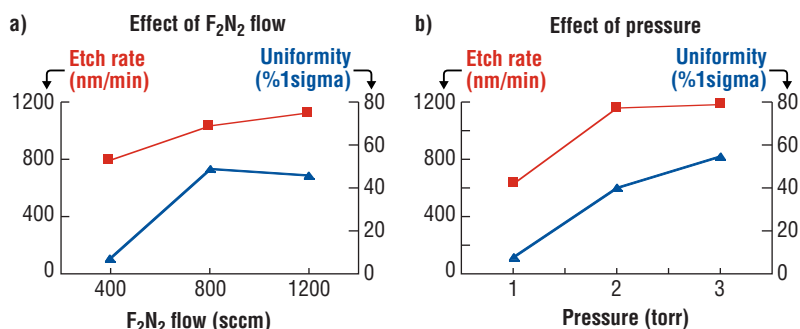


Figure 1. Taguchi-L9 matrix of experiment of SiO<sub>2</sub> etch rate (nm/min) (■) and etch non uniformity (▲) (% 1σ) of N<sub>2</sub>/F<sub>2</sub> gas as a function of a) flow rate, and b) pressure. The SiO<sub>2</sub> layer thickness is 1μm.

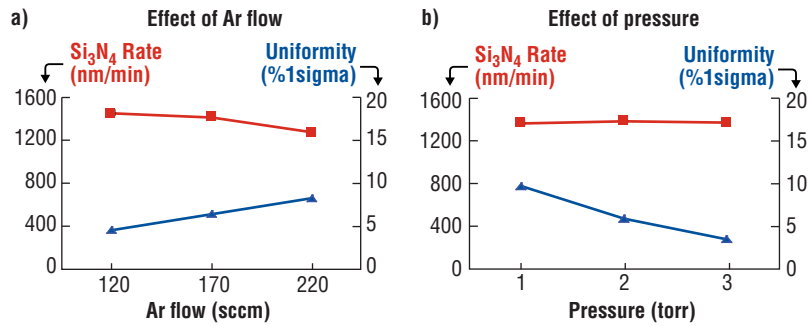
## Experimental

The evaluation of the F<sub>2</sub> gas mixtures was performed on 200mm wafers in an Applied Materials PECVD reactor on a P5000 main frame. The F<sub>2</sub>-containing gas bottle was installed inside a gas cabinet close to the CVD reactor. For the safe, clean and dry handling of the fluorine gases, a 3-way purging unit was part of the gas supply. The gas line was connected using a T-connection to the existing standard CF<sub>4</sub> gas line, which is used as the standard Applied Materials chamber cleaning gas for this reactor. For precise gas flow control, a metal-sealed MFC was applied. Two F<sub>2</sub> resistant O-rings (Isolast 9675) from Trelleborg Sealing Solution Germany GmbH were installed: one for the chamber lid outer seal, and one for the gas feed-through.

According to the Solvay-procedure regarding safety and stainless steel passivation with F<sub>2</sub>, a helium leak test, as well as a vacuum leak check for the relevant gas line, were done. N<sub>2</sub>F<sub>2</sub> gas was then flowed for >10hrs to passivate the stainless steel surface.

Based on a former evaluation by Solvay, as well as Applied Materials' BKM (best-known method) recipes, the first plasma ignition of N<sub>2</sub>/F<sub>2</sub> gas was achieved immediately.

The best mixture and the best working parameters were selected calculating three L9-Taguchi matrices. The best mixture was identified



**Figure 2.** Taguchi-L9 design of experiment of Si<sub>3</sub>N<sub>4</sub> etch rate [nm/min] and etch non uniformity (% 1 $\sigma$ ) of Ar/N<sub>2</sub>/F<sub>2</sub> gas mixture as a function of a) Ar-flow, and b) pressure.

to be the following composition: 10% Ar/ 70% N<sub>2</sub>/ 20%F<sub>2</sub>.

To investigate the etching gas performance, SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub> layers were used. Blank 200mm Si-wafers were deposited with a 1 $\mu$ m-thick oxide layer (silane-based PECVD); additionally, Si wafers were deposited with a 550nm-thick Si<sub>3</sub>N<sub>4</sub> film.

As a complementary part of this work, the etch rate of the selected F<sub>2</sub> mixture on amorphous silicon (a-Si) was measured. A layer of 300nm (the maximum allowed by this equipment) of a-Si was deposited on a layer of 100nm SiO<sub>2</sub>, which is the necessary substrate for thickness measurements.

To compare the etch performance of Ar/N<sub>2</sub>/F<sub>2</sub> on SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub>, respectively, tests using standard CVD cleaning gases, such as NF<sub>3</sub>, CF<sub>4</sub> and C<sub>2</sub>F<sub>6</sub>/O<sub>2</sub>, were done. The CF<sub>4</sub>-based BKM chamber cleaning gas is recommended by Applied Materials. A further L9-Taguchi matrix was evaluated to define the best valid recipe for NF<sub>3</sub>.

Three repeatability runs with the selected 10%Ar/70%N<sub>2</sub>/20%F<sub>2</sub> mixture were performed using a batch of 25 wafers/run. The wafers in slots 1, 12, and 25 were monitored for particle contamination. The particle performance behavior of the cleaning gas mixture was measured on particle monitor wafers by a Tencor Surfscan 6400. The minimal particle size measured was 0.25 $\mu$ m.

## Results

The first plasma ignition of the F<sub>2</sub>/N<sub>2</sub> mixture could be achieved without observing arcing or other unknown effects, with the following parameters: Chamber pressure: 5Torr; N<sub>2</sub>/F<sub>2</sub> gas flow rate of 1slm, temperature of 400 °C and 570 mils spacing.

The effects of the parameter variation on the performance of the F<sub>2</sub>/N<sub>2</sub> plasma were evaluated through a first L-9 Taguchi matrix on SiO<sub>2</sub>. As seen in Fig. 1, there is a significant correspondence between the gas flow and chamber pressure to the achievable etch rate. The increase of the etch nonuniformity, is the limiting factor to improve etching performance.

The cleaning gas behavior on Si<sub>3</sub>N<sub>4</sub> with the addition of Ar at the N<sub>2</sub>/F<sub>2</sub> mixture was studied next (Fig. 2). A preliminary Taguchi matrix study evidenced a reduced dependency of the etching performance on the RF power applied. A small addition of Ar notably increases the uniformity of the etching.

It is possible to achieve etching rates >1300nm/min even while maintaining nonuniformity values <5%, far below the value of 20%,

which is generally considered the highest acceptable. These results are more encouraging because the addition of Ar to the F<sub>2</sub>/N<sub>2</sub> mixture dilutes the concentration of the active specie F<sub>2</sub>, which is not desirable. Figure 2a shows the slight decrease in etch rate with increasing Ar-flow.

As a consequence of the described test results, the mixture 10% Ar / 70% N<sub>2</sub> / 20% F<sub>2</sub>, was chosen for the next parts of this study. The mixture was premixed and delivered in a cylinder to avoid any F<sub>2</sub> dilution effect by adding Ar during operation.

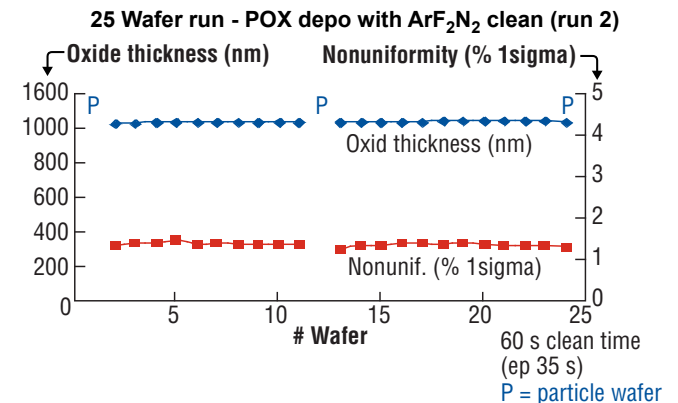
To test repeatability within wafer, wafer-to-wafer, and batch-to-batch of the Ar/N<sub>2</sub>/F<sub>2</sub> etching and particle performance, three runs of 25 wafers each were performed. Processing the wafer batch was done in full automatic mode by the deposition of a 1 $\mu$ m SiO<sub>2</sub> film for 35 sec. and a subsequent 60 sec. plasma chamber clean in Ar/N<sub>2</sub>/F<sub>2</sub> gas. The particle monitor wafers were placed in slots 1, 12 and 25, respectively. Immediately after SiO<sub>2</sub> deposition, the wafers left the CVD reaction chamber.

**Table 1.**

Run #	Depo nonuniformity		Clean nonuniformity		
	SiO <sub>2</sub> thick nm	Within waf 1% sigma	Waf-waf 1% sigma	Clean rate nm/min	Within waf 1% sigma
1	1040	1.2	0.6	n/m	n/m
2	1034	1.4	0.8	1525	7.1
3	1035	1.3	0.6	1518	8.8
<b>Mean</b>	<b>1036</b>	<b>1.3</b>	<b>0.7</b>	<b>1522</b>	<b>8.0</b>

Summary of the three repeatability runs of 1 $\mu$ m-SiO<sub>2</sub> deposition for 35 sec, followed by an Ar/N<sub>2</sub>/F<sub>2</sub> chamber clean etching for ~60 sec.

The cleaning recipe selected was a gas flow rate of 900 sccm Ar/N<sub>2</sub>/F<sub>2</sub> and a chamber pressure of 2.1 Torr. The RF power was 800W and the susceptor temperature 400°C with 540 mils spacing (maximum spacing value on the silane-based oxide/nitride CVD chamber).



**Figure 3.** Repeatability (wafer-to-wafer) runs of 1 $\mu$ m-SiO<sub>2</sub> deposition for 35 sec, followed by an Ar/N<sub>2</sub>/F<sub>2</sub> chamber clean etching for 60 sec (run 2). The graph displays the oxide deposition thickness (◆) (nm/min) and oxide deposition non uniformity (■) (% 1 $\sigma$ ) as a function of wafer position (total 25 wafers). The particle monitor wafers (P) are located at the wafer position 1, 12, and 25.

The total cleaning time of 60 sec., included an over-cleaning of ~30% to make sure any previously deposited oxide at the chamber walls, pumping plates, susceptor edges, and on the shower head will be completely removed.

The deposition recipe to deposit 1µm of SiO<sub>2</sub> was the standard BKM recipe of Applied Materials with SiH<sub>4</sub> and N<sub>2</sub>O as the main process gas.

Figure 3 shows the results of repeatability runs of 1µm SiO<sub>2</sub> deposition followed by an Ar/N<sub>2</sub>/F<sub>2</sub> chamber clean etch (run 2). The graphs display the oxide deposition thickness as well as the oxide deposition nonuniformity as a function of wafer position.

A SiO<sub>2</sub> deposition of ~1034nm was achieved for all 25 wafers. The oxide deposition nonuniformity was measured within-wafer as well as wafer-to-wafer. The within-wafer oxide deposition nonuniformity was ~±1.4% (1σ) and wafer-to-wafer nonuniformity was ±0.8% (1σ), demonstrating a very stable oxide deposition.

To verify the repeatability of the SiO<sub>2</sub> oxide etching rate for 25 wafers, the previously deposited wafers were run again and partially etched by Ar/N<sub>2</sub>/F<sub>2</sub> for 30 sec. After measuring the remaining oxide thickness, the cleaning rate was calculated.

Figure 4 shows the results of SiO<sub>2</sub> etching rate of Ar/N<sub>2</sub>/F<sub>2</sub> as a function of wafer position (run #2). For the first wafer, the etch rate was ~1280nm/min and significantly lower compared to the rest of the 24 wafer batch, indicating the “first wafer effect.” From the second wafer on, the oxide etching rate averaged ~1525nm/min for all wafers with a corresponding within-wafer oxide etch nonuniformity of ±7.1% (1σ). This shows, within wafer, as well as wafer-to-wafer, a very repeatable and stable oxide etching rate.

Table 2.

Run #	# adders (particle size > 25 µm)			
	Slot 1	Slot 12	Slot 25	Average
1	38	7	21	22
2	5	5	32	14
3	11	2	6	6
<b>Mean</b>				<b>14</b>

The number of particle adders with a particle size ≥0.25µm on 200mm (slot 1, 12 and 25). The SiO<sub>2</sub> deposition (1036nm for 35sec.) was followed by a subsequent Ar/N<sub>2</sub>/F<sub>2</sub> oxide etch (>1500nm for 60 sec.) for three wafer runs (total 75 wafers).

To verify Ar/N<sub>2</sub>/F<sub>2</sub> etch rate nonuniformity on SiO<sub>2</sub> from batch-to-batch, three wafer runs (75 wafers) were carried out. Table 1 displays the result of all three runs. An average SiO<sub>2</sub> deposition thickness of 1036nm was achieved for all three batches. For all three runs, the within-wafer oxide deposition nonuniformity averaged ±1.3% (1σ) and the wafer-to-wafer nonuniformity averaged ±0.7% (1σ), demonstrating a constant oxide

deposition across all three batches. The data shows the average values of SiO<sub>2</sub> layer deposition thickness (nm), oxide deposition nonuniformity (within wafer and wafer-to-wafer at %1σ) and Ar/N<sub>2</sub>/F<sub>2</sub> oxide etching rate (nm/min) nonuniformity within wafer (%1σ) for three wafer runs (total 75 wafers). The average (mean) values of the batch-to-batch uniformity are shown as well. For the first run, the etch rate was not measured.

The Ar/N<sub>2</sub>/F<sub>2</sub> etching rate on SiO<sub>2</sub>, batch-to-batch, was on average (mean) 1522 nm/min with a corresponding within-wafer oxide etch nonuniformity of ±8.0% (1σ). This demonstrates a very repeatable and stable oxide etching rate of the Ar/N<sub>2</sub>/F<sub>2</sub> gas mixture for all wafer runs.

To get information about the particle performance behavior of the Ar/N<sub>2</sub>/F<sub>2</sub> etching gas mixture on the wafer surface, the three particle monitor wafers were measured before and after the wafer run (Table 2). Using a Tencor Surfscan 6400, the minimal particle size measured was 0.25µm. The virgin reference wafer showed a particle contamination of ~20 particles (0.25µm).

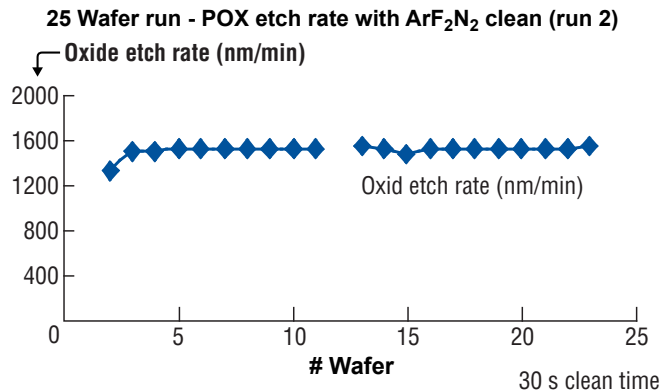
Table 3.

Cleaning gas	SiO <sub>2</sub>		Si <sub>3</sub> N <sub>4</sub>		Total	Clean gas	Clean gas	Net	Pressure	RF-power	Sucs.
	Etch rate	Unif	Etch rate	Unif	Flow	Flow	Flow	F-amount			Spacing
	[nm/min]	[%] 1 sigma	[nm/min]	[%] 1 sigma	[sccm]	[sccm]	[gr/min]	[g/min]	[Torr]	[W]	[mils]
ArN <sub>2</sub> F <sub>2</sub>	1521	12	1950	8	900	180	0.31	0.00	2.1	800	570
CF <sub>4</sub>	850	2	1710	9	2005	1550	6.09	0.00	5.5	1000	570
NF <sub>3</sub>	<1200	20	<1570	20	150	150	0.48	0.00	0.6	600	570
C <sub>2</sub> F <sub>6</sub> /O <sub>2</sub> /NF <sub>3</sub> (Average)	1116	10	1303	40							
C <sub>2</sub> F <sub>6</sub> /O <sub>2</sub> /NF <sub>3</sub> (Outer clean)	439	11	716	47	1560	750/60	4.62/0, 19	3.96	1.8	900	1000*
C <sub>2</sub> F <sub>6</sub> /O <sub>2</sub> (Inner clean)	1792	8	1890	33	1850	900	5.54	4.58	9.0	900	200
NF <sub>3</sub>	932	9	1300	20	140	140	0.44	0.36	0.6	350	800**

Amount of cleaning gas and cleaning time needed for three different cleaning gas chemistries (Ar/N<sub>2</sub>/F<sub>2</sub>, CF<sub>4</sub>, C<sub>2</sub>F<sub>6</sub>/O<sub>2</sub> and NF<sub>3</sub>) normalized to 1µm dielectric film (SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub>) thickness (20% over-cleaning time included).

After SiO<sub>2</sub> deposition and oxide etching, the average particle adders on the wafer surface was 22 particles for run 1, 14 particles for run 2, and 6 particles for run 3. On average (mean), just 14 particle adders were generated from run-to-run. The data indicate that no significant particle contamination was induced by the process.

All the obtained oxide etch rates and oxide etch nonuniformities of the Ar/N<sub>2</sub>/F<sub>2</sub> cleaning gas mixture on SiO<sub>2</sub> are equal



**Figure 4.** Repeatability (within-wafer and wafer-to-wafer) runs of SiO<sub>2</sub> etching rate (nm/min) of Ar/N<sub>2</sub>/F<sub>2</sub> for 30 sec. as a function of wafer position (total of 25 wafers).

to or better than the standard CF<sub>4</sub> based BKM chamber clean of Applied Materials. The cleaning time achieved with the Ar/N<sub>2</sub>/F<sub>2</sub> gas mixture was ~30% shorter than the CF<sub>4</sub>-based BKM Applied Materials clean time. The particle values were <50 adders, which is well within specification for the AMAT P5000 process based on CF<sub>4</sub> cleaning chemistry.

The chamber was opened after processing the first 25 wafer run, as well as after the final run (run 3), and visually inspected. For both inspections, the surface of the shower head, shadow ring, pumping plate and chamber lid O-rings, were clean and showed no signs of wear or residues.

Si-pieces with a thin SiO<sub>2</sub> layer were placed on the pumping ring to check the cleaning efficiency outside the susceptor area. The complete oxide layer on the Si-pieces was removed throughout the three repeatability runs, which verifies that the Ar/N<sub>2</sub>/F<sub>2</sub> gas mixture cleaned all important CVD chamber areas very well.

With the aim to achieve a direct performance comparison between the selected F<sub>2</sub> mixture and NF<sub>3</sub>, a new L9 Taguchi matrix was calculated on SiO<sub>2</sub> deposited layers to select the best working parameters for NF<sub>3</sub> in the oxide/nitride chamber.

NF<sub>3</sub> flow rates >150sccm and a chamber pressure >0.6Torr cause nonuniformity >20%, which is not acceptable for a proper chamber cleaning. The maximum achievable etching rate with a nonuniformity <20% (1 $\sigma$ ) was 1200nm/min. **Table 3** summarizes all obtained etch rates for SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub>.

To confirm the reliability of the obtained data on the oxide/nitride chamber, the etch rate on SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub> was measured in a tungsten chamber, where NF<sub>3</sub> is a standard installation. For this chamber type, AMAT suggests a BKM chamber cleaning recipe. Its etching performance does not differ appreciably from the best parameter we set on

the reference oxide/nitride chamber. It can be supposed that an F<sub>2</sub> mixture can also improve cleaning performance in a TEOS chamber that normally uses a C<sub>2</sub>F<sub>6</sub>/O<sub>2</sub>/NF<sub>3</sub> cleaning mixture, although no direct tests have been performed.

The total cost of a chamber cleaning process is determined from the wafer throughput of the CVD equipment and the cost of the consumables. Owing to the high cost of the equipment, the throughput is considered to have the highest impact. Using the etching rates of the different gas recipes in **Table 4**, it is possible to estimate the required time and amount of gas to etch, for example, 1 $\mu$ m of SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub>. The required cleaning time is directly correlated to the throughput of the equipment, while the amount of required gas directly influences the cost of consumables.

The tested mixture Ar/N<sub>2</sub>/F<sub>2</sub> showed improvements in both parameters, cleaning at a faster rate, and even requiring a lesser amount of gas. In particular, the NF<sub>3</sub> cleaning method for SiO<sub>2</sub> processes requires a longer cleaning time (+27%) and almost double the amount of process gas (+196%).

A layer of 300nm of a-Si was deposited on a 100nm layer of SiO<sub>2</sub>. Our standard Ar/N<sub>2</sub>/F<sub>2</sub> mixture was applied for 15 sec., setting the plasma parameters at optimum conditions. The F<sub>2</sub> cleaning recipe was allowed to etch both the a-Si layer and the SiO<sub>2</sub> substrate completely and uniformly, resulting in an etching rate >1200nm/min. The test was repeated with the NF<sub>3</sub> best cleaning recipe that was defined in this work. The a-Si layer was neither completely, nor uniformly etched.

## Conclusion

The selected Ar/N<sub>2</sub>/F<sub>2</sub> mixture can be applied as a drop-in replacement to the existing cleaning chemistries. The tests have been carried out on an industrial tool – an AMAT P5000 – which was demonstrated to be fully compatible with the new cleaning gas. The F<sub>2</sub> gas line was connected to the existing CF<sub>4</sub> gas line by applying a 3-way purging unit. The F<sub>2</sub> mixture is compatible with stainless steel if a proper passivation procedure is performed.

The high etching rate and the low gas requirement of the selected Ar/N<sub>2</sub>/F<sub>2</sub> mixture indicate that a significant cost-of-ownership reduction can be achieved. The cleaning recipes that have been taken as reference are a) the BKM CF<sub>4</sub>- cleaning recipe from AMAT and b) an NF<sub>3</sub>-based cleaning recipe that has been selected and optimized during the tests.

The global warming potential of the selected Ar/N<sub>2</sub>/F<sub>2</sub> mixture is zero. This can contribute to the achievement of the greenhouse emissions reduction targets of the semiconductor industry. ■

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Table 4.

Cleaning gas	Layer Thickness ( $\mu\text{m}$ )	Cleaning Time [sec]	$\text{SiO}_2$		$\text{Si}_3\text{N}_4$		
			Required gas [gr]	Required gas % vs. $\text{F}_2$	Cleaning Time [sec]	Required gas [gr]	Required gas % vs. $\text{F}_2$
$\text{ArN}_2\text{F}_2$	1.0	47.3	0.245	100%	36.9	0.191	100%
$\text{CF}_4$	1.0	84.7	8.598	3515%	42.1	4.274	2240%
$\text{NF}_3$	1.0	60.0	0.480	196%	45.9	0.367	192%

Comparison of the required  $\text{F}_2$  gas consumption [%] for  $\text{Ar}/\text{N}_2/\text{F}_2$ ,  $\text{CF}_4$  and  $\text{NF}_3$  on  $\text{SiO}_2$  and  $\text{Si}_3\text{N}_4$ , respectively.

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